X-ray Imaging Techniques, Applications and Detectors

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Outline

- Techniques and Applications
- Detectors
- Role of DOE labs

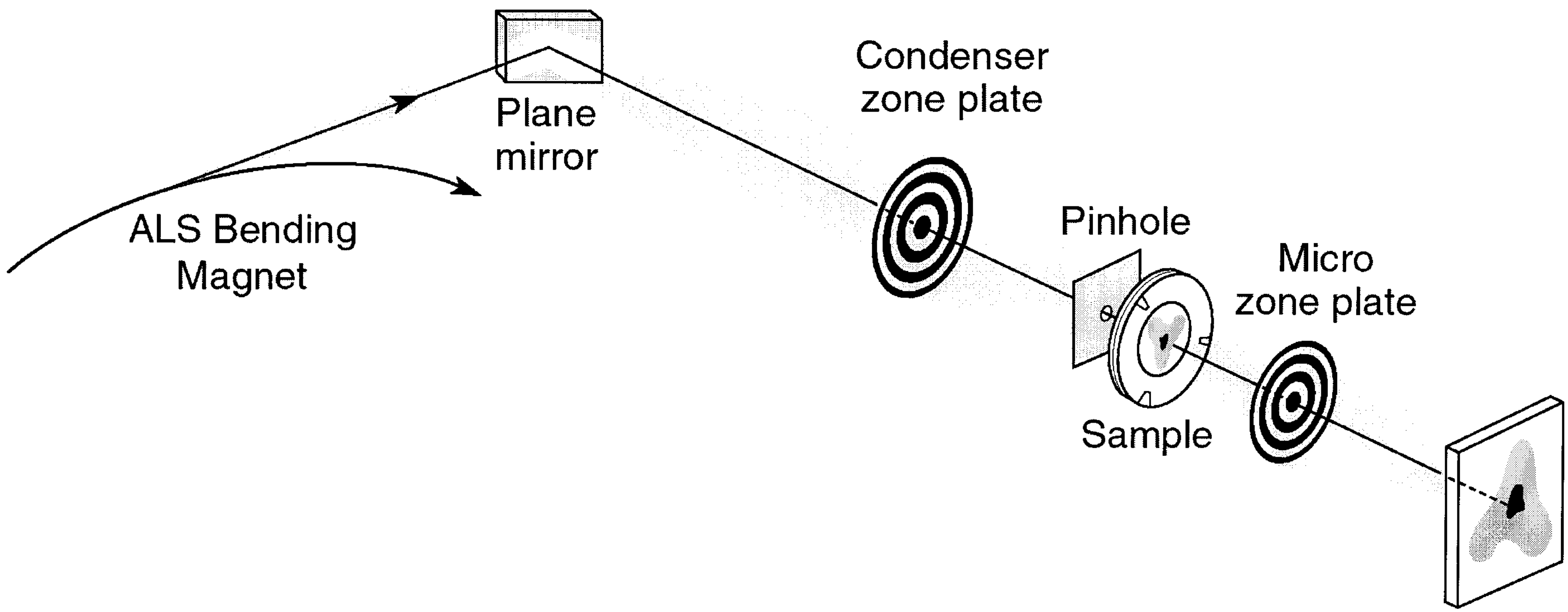
Perspective

- Run a national facility for earth-science research at APS.
- Techniques include diffraction, microXRF, spectroscopy, tomography
- Many detectors in use on the sector, all commercial
- 7 CCDs
  - 2 Bruker Smart systems for diffraction
  - 2 Roper systems for microtomography (lens-coupled)
  - 3 Roper visible light systems for emission spectroscopy and Raman scattering
- 3 multi-element Ge detectors for x-ray absorption spectroscopy and energy-dispersive diffraction
- Microspec (Oxford) wavelength spectrometer for high-energy resolution spectroscopy

X-ray Imaging Techniques and Applications

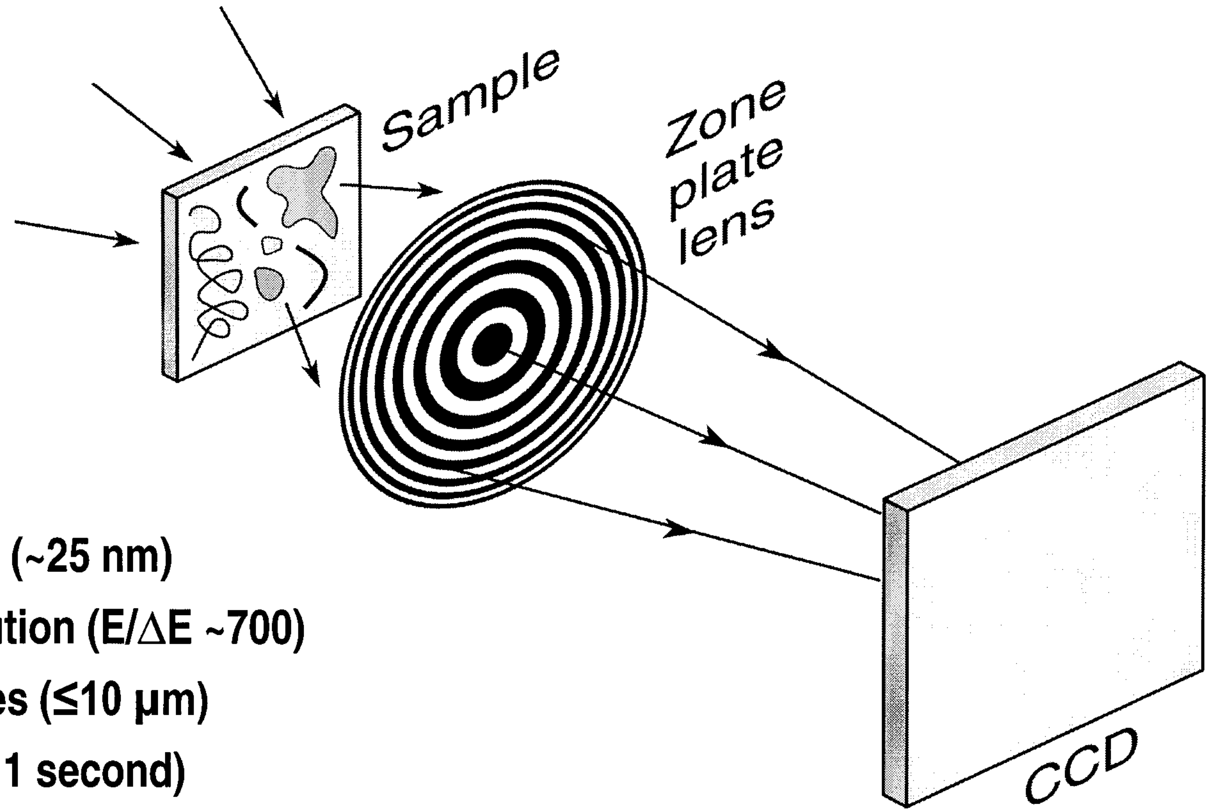
- Scanning soft x-ray microscopy
- Projection soft x-ray microscopy
- Radiography
- Absorption tomography
- Phase-contrast tomography
- Holography
- Diagnostics (focusing aid, particle beam size/position monitors)
- Scanning fluorescence techniques (not covered here, detectors are the same as EXAFS covered by others).

The XM-1 Soft X-ray Microscope at the Advanced Light Source



E = 250eV - 900eV
λ = 1.4nm - 5nm

X-Ray Microscopy at the ALS

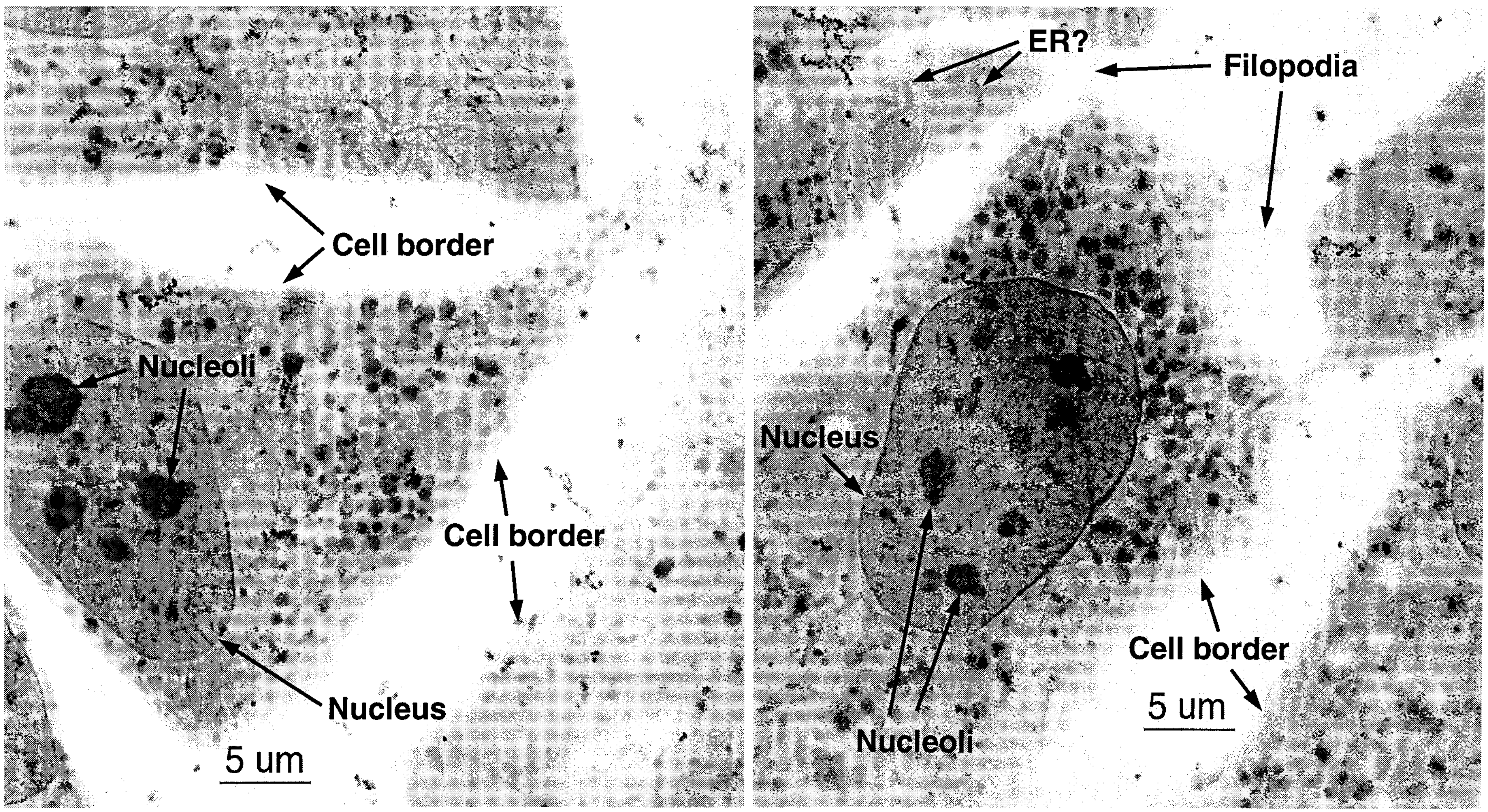


- High spatial resolution (~25 nm)
- Modest spectral resolution (E/ΔE ~700)
- Hydrated, thick samples (≤10 µm)
- Short exposure time (~1 second)
- Mutually indexed visible and x-ray microscopes
- High throughput (up to a thousand images per day)
- Large image fields by montage assembly
- Easy access, user friendly

Organelle Details Imaged with Cryogenic Preservation and High Spatial Resolution

BERKELEY LAB

Cryo x-ray microscopy of 3T3 fibroblast cells



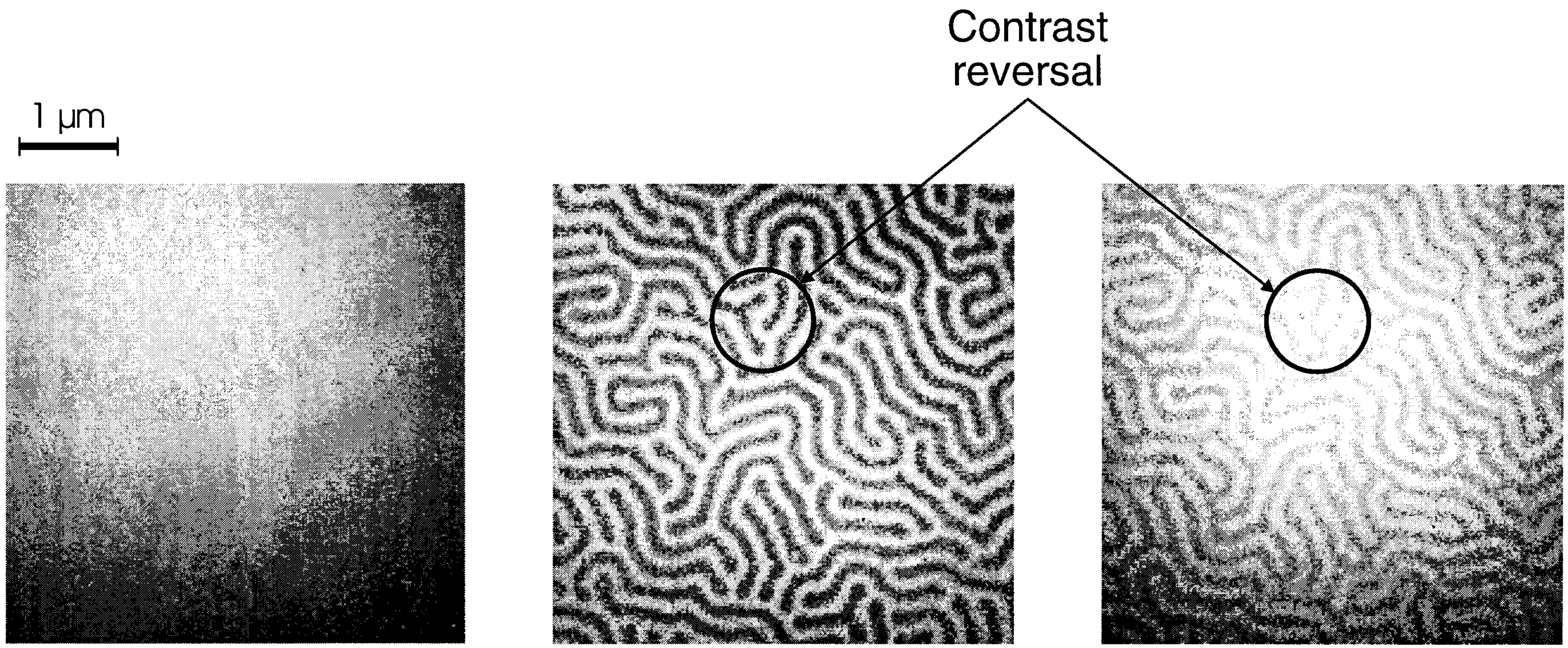
CXRO
C. Larabell, D. Yager, D. Hamamoto, M. Bissell, T. Shin / LBNL Life Sciences Division
W. Meyer-Ilse, G. Denbeaux, L. Johnson, A. Lucero / CXRO

Magnetic X-Ray Microscopy

- High spatial resolution in transmission
- Bulk sensitive (thin films)
- Complements surface sensitive PEEM
- Good elemental sensitivity
- Allows applied magnetic field
- Insensitive to capping layers

Magnetic Domains Imaged at Different Photons Energies

FeGd Multilayer



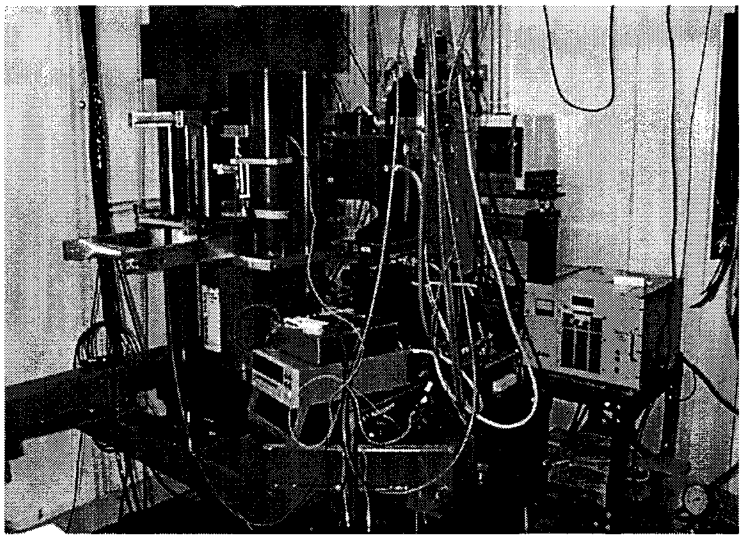
$\hbar\omega = 704$ eV
below Fe L-edges

$\hbar\omega = 707.5$ eV
Fe L_3-edge

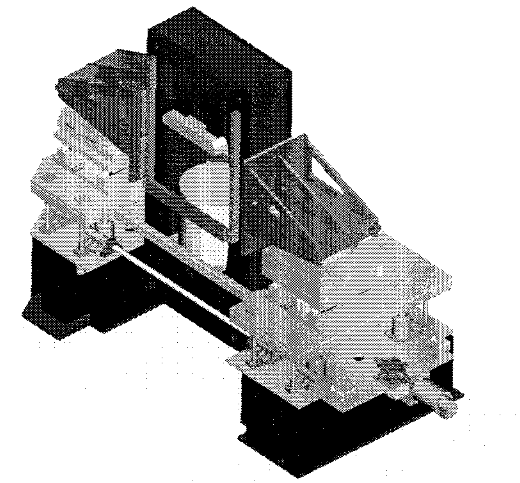
$\hbar\omega = 720.5$ eV
Fe L_2-edge

P. Fischer, T. Eimueller, M. Koehler / U. Wuerzberg
S. Tsunashima / U. Nagoya and N. Tagaki / Sanyo
G. Denbeaux, L. Johnson, A. Lucero / CXRO

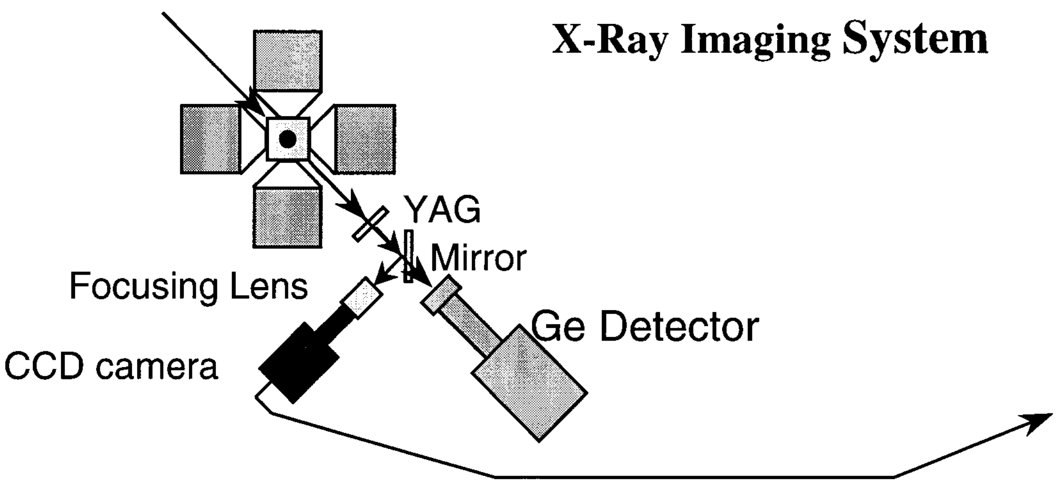
250 T LVP: BM Line

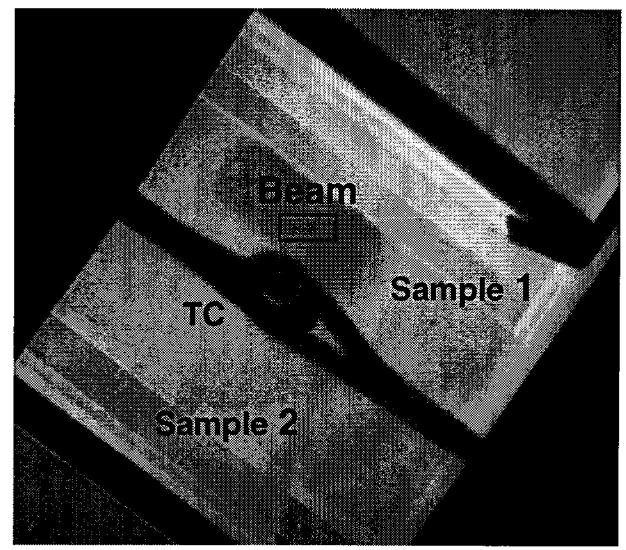


1000 T LVP: ID Line



X-Ray Imaging System





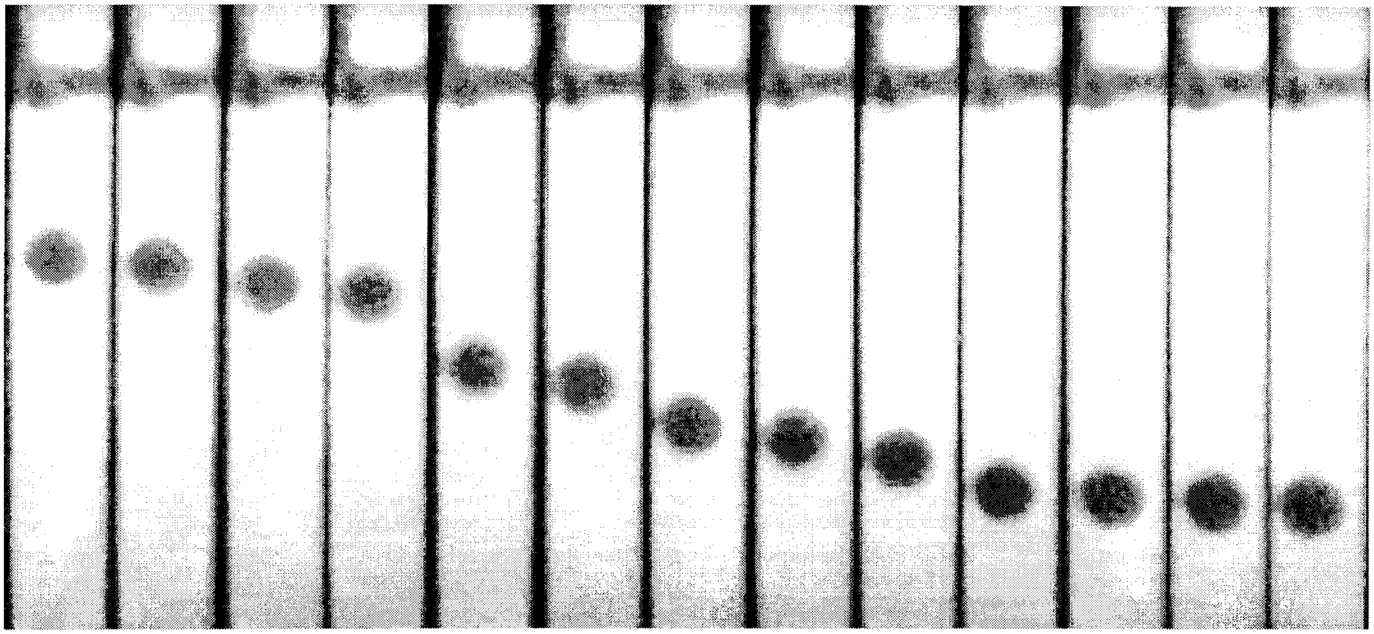


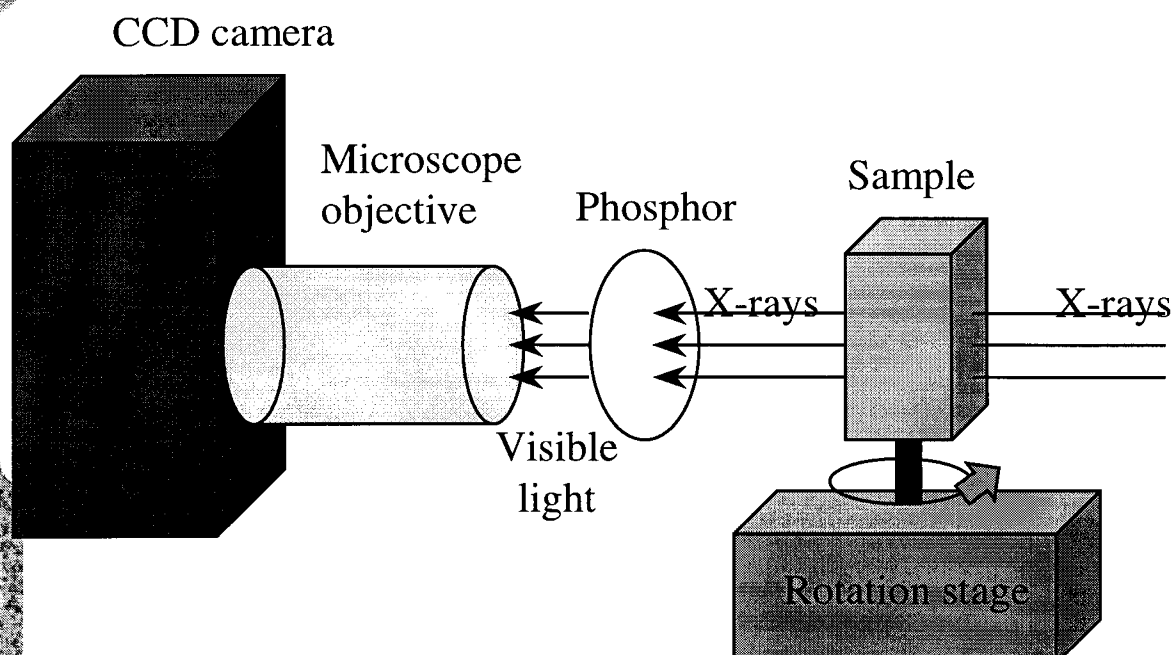
Fig. LVP9. X-ray radiograph snapshots of a 0.3 mm diameter W sphere falling in sodium disilicate melt at 2.6 GPa and 1400C. Gaps between anvils about 0.5 mm. Sample was in a Pd capsule; top portion of the capsule can be seen as the dark contrast near the top of each snapshot. Radiographs captured by a CCD camera. Experiments performed by Reid et al. (1999) at GSECARS at the APS (13-BM-D) using white beam and the DIA apparatus.

Microtomography

- Allows study of internal structure of objects which cannot be sectioned
  - Too valuable
  - Too fragile
  - Too time-consuming
- Geoscience applications
  - Fractures and pores
    - Fluid transport
    - Waste encapsulation
    - Hydrocarbon reservoirs
- Mineral inclusions
  - High-pressure phases in diamonds
- Fossil morphology
- Others
  - Materials sciences, ceramics, etc.

Computed Microtomography

- Like medical CAT scanners, but with micron resolution
- Construct cross-sections of an object mathematically from multiple projections as object is rotated

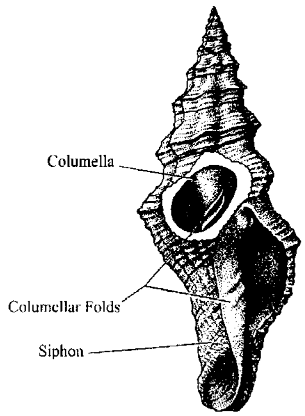


Tomography of Fossils

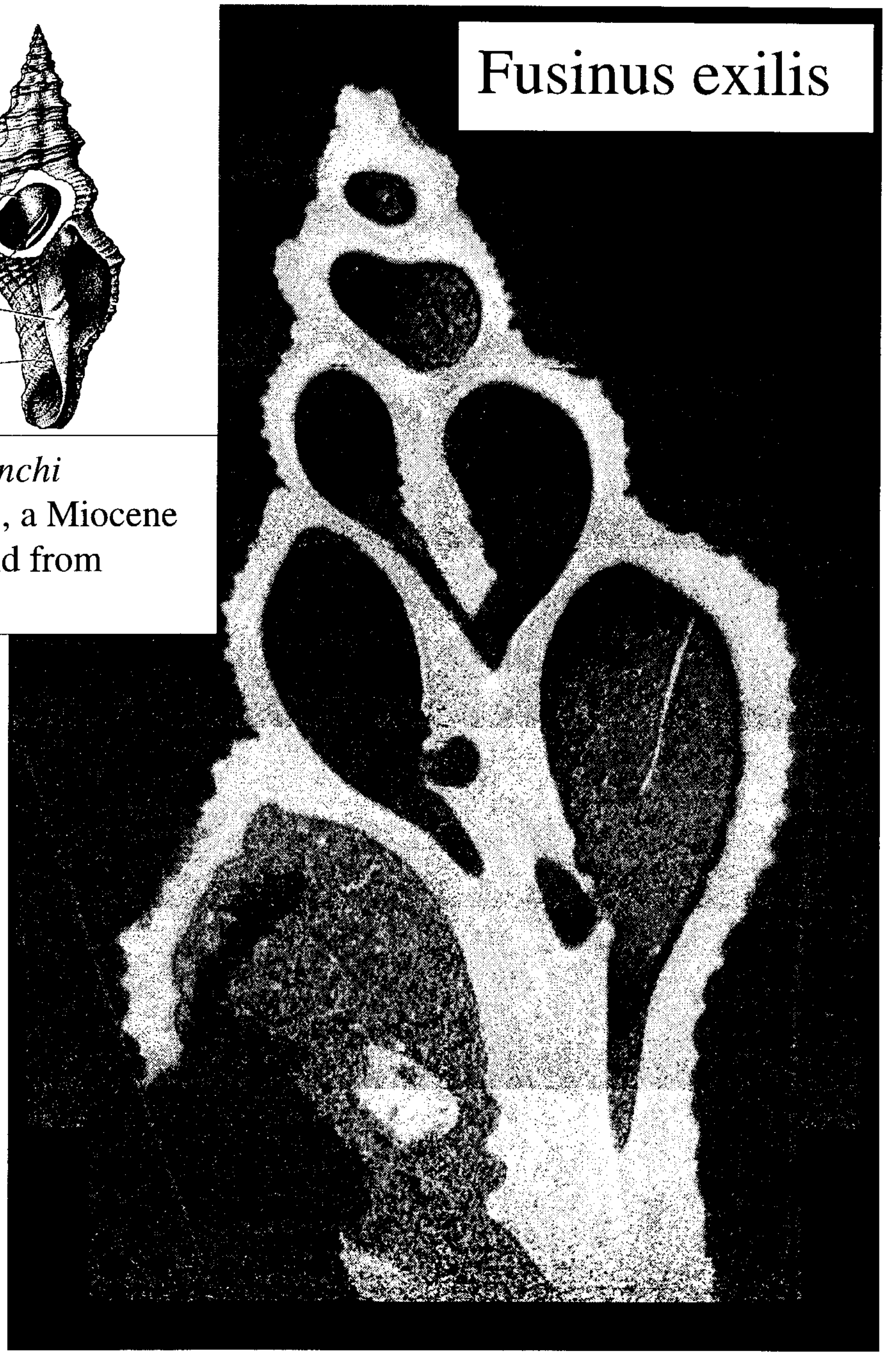
- Many fossils are rare and cannot be physically sectioned
- Want to study internal morphology to understand function and evolution
- Need high-energy x-rays
- These images were collected at 40 keV with temporary single-bounce monochromator

Evolution of Marine Snails

(B. Price, M. Rivers; U. of Chicago)



Latirus lynchi (Basterot), a Miocene fasciolariid from France



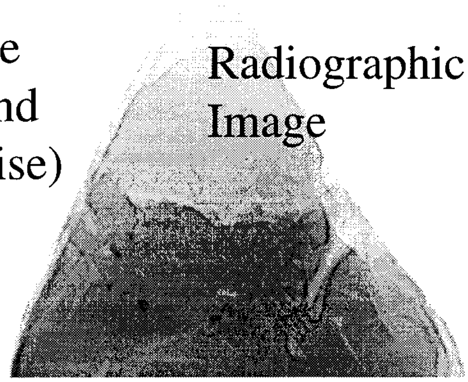
Tomography of Diamonds

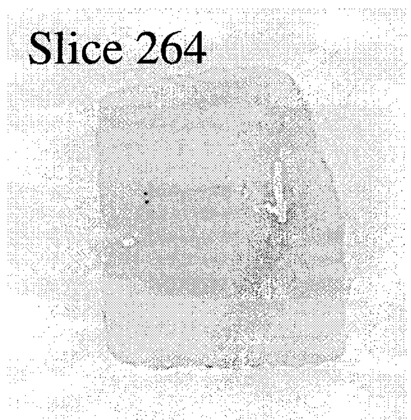
- Want to study inclusions in diamonds
- These inclusions are among the deepest samples of earth materials which are available
- Deepest diamonds are opaque
- Use tomography to locate inclusions, then use diffraction and spectroscopy to study them in-situ

Mineral Inclusions in Opaque Diamonds

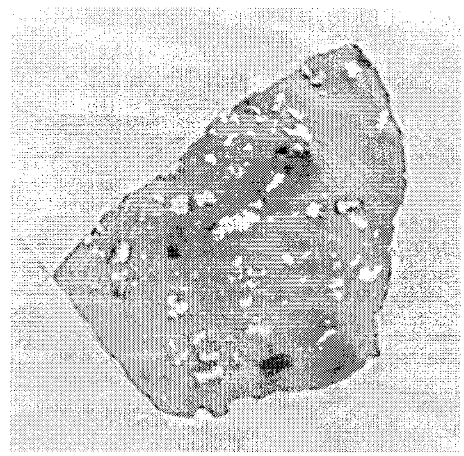
(M. Rivers, U. of Chicago)

Opaque diamond (J. Parise)



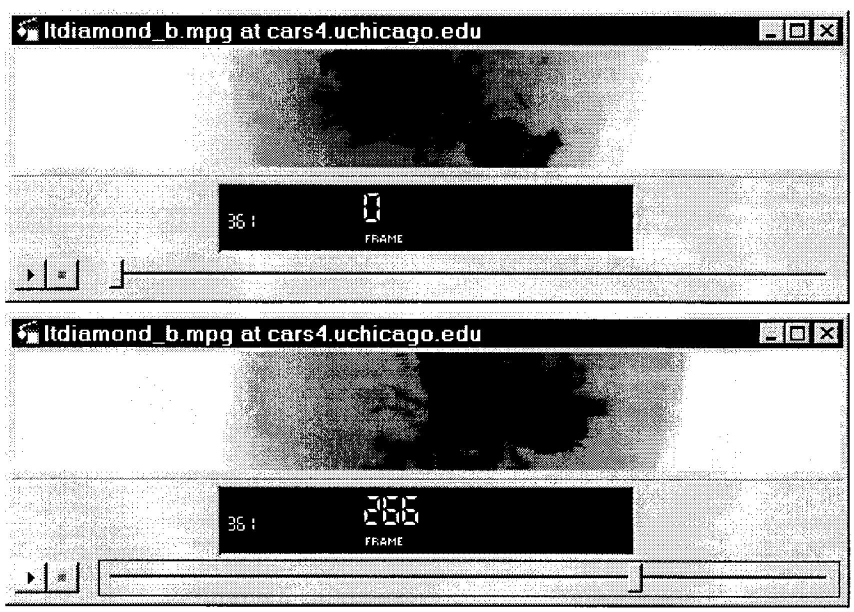


Carbonado diamond (E. Vicenzi, Princeton U.)



22 karat diamond with massive dendritic sulfide inclusion

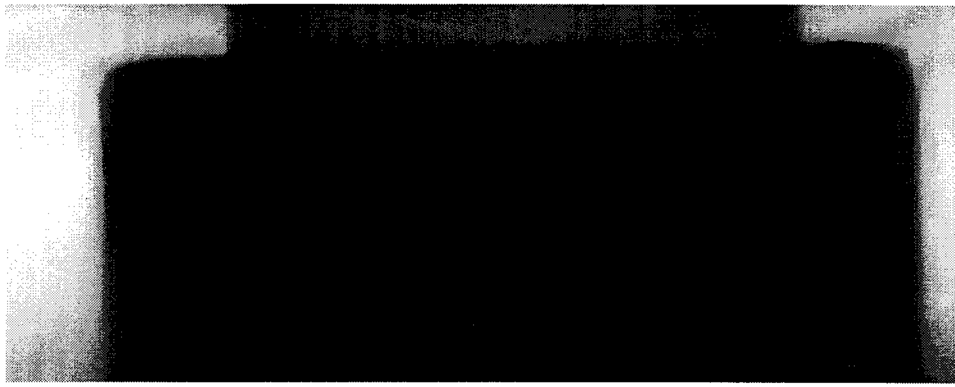
(L. Taylor, U. Tennessee)



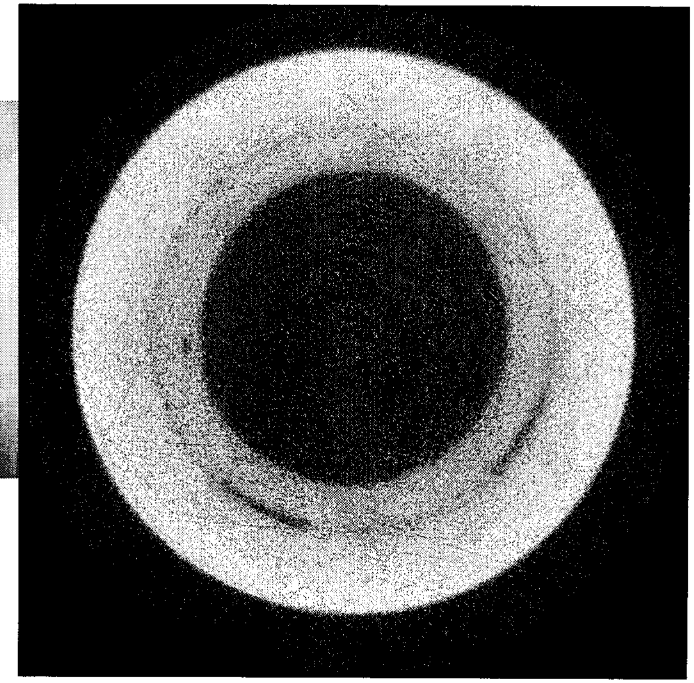
Microtomography at 80 keV

- Studying Sr-Fe-Co oxide ceramics used for oxygen gas permeability
- 1 cm diameter, requires 80 keV
- Absorption depth in YAG phosphor crystal is > 1mm, need thick crystal. Y Ka fluorescence travels >100 microns, hard to get good spatial resolution
- Pixel size is 10 microns, resolution is >50 microns

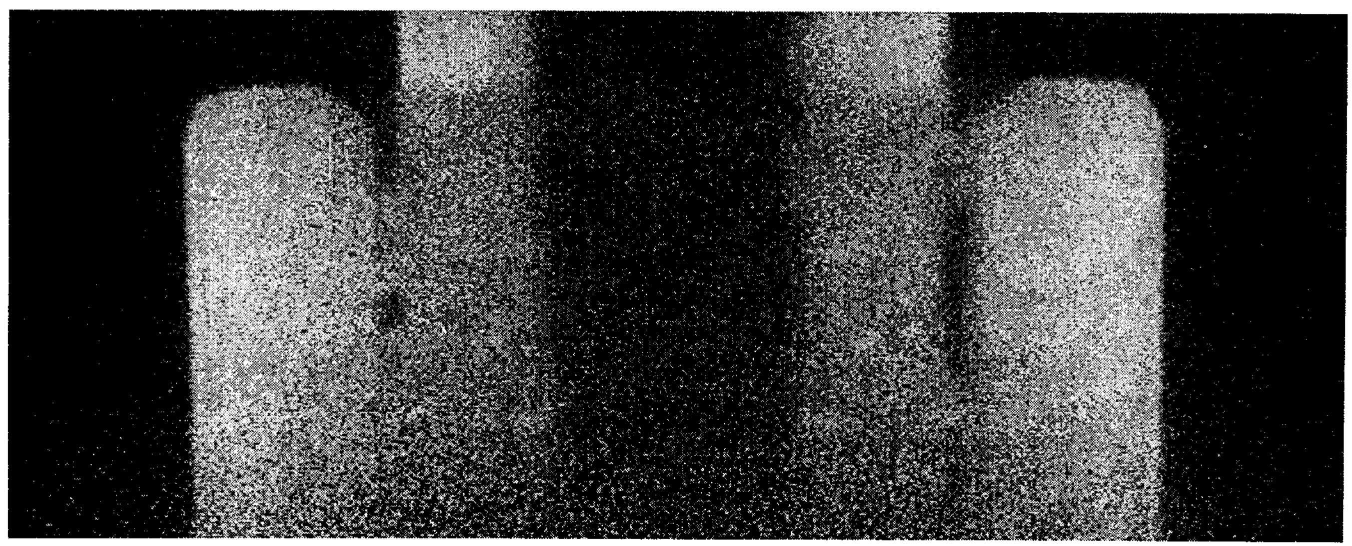
Radiograph



Z-slice

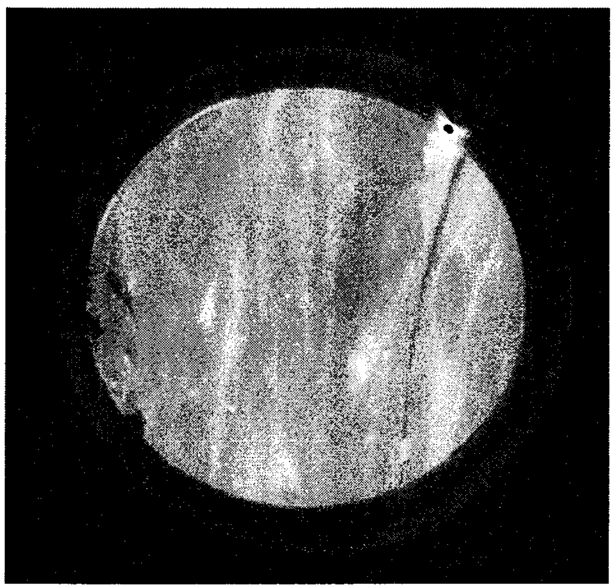


X-slice

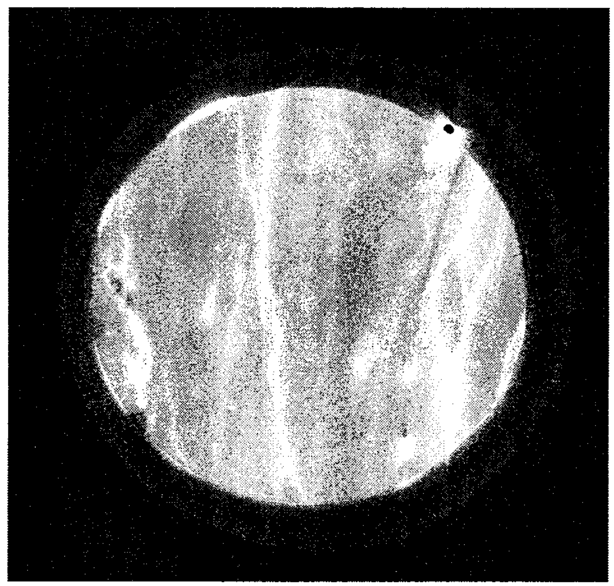


Contrast enhancement by digital subtraction tomography

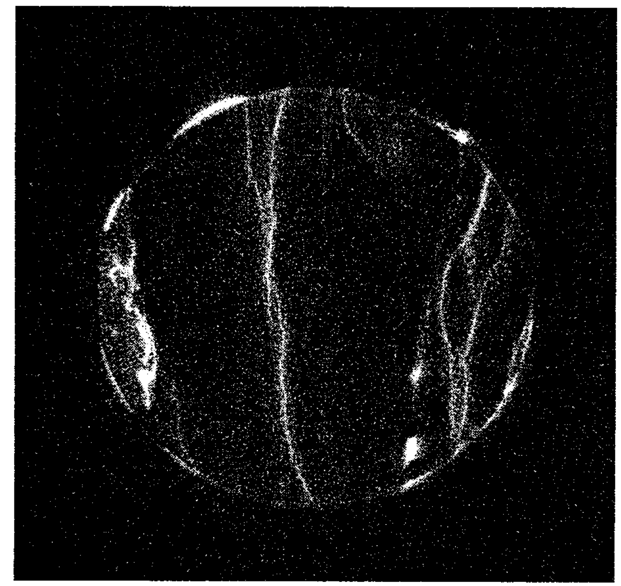
This 1cm diameter rock core was saturated with CsCl solution and images were taken above and below the Cs K absorption edge (36 keV). The difference image shows only the solution, which is present in the cracks.



36.0 keV, below Cs K absorption edge



36.2 keV, above Cs K absorption edge



Difference of above and below edge images

Detectors for Full-Field Imaging

- CCD detector is the workhorse.
- Positive characteristics
  - < 10 electron read noise
  - Negligible dark current when cooled below –25C
  - High resolution (>1Kx1K)
  - Relatively rapid readout (.5-1MHz @16bits, 5-20 MHz @12 bits)
  - 16 bit dynamic range for large pixels, 12-14 bits for small pixels, determined by full-well capacity
  - Reasonable sensitivity from NIR to NUV

Deficiencies of CCDs for x-ray imaging: X-ray Coupling

- Direct illumination @10 keV
  - Thin Si is transparent, poor absorption efficiency
  - ~2 eV per electron/hole pair, 5000 electrons per x-ray
  - Full well capacity is only 50,000-500,000 electrons, = 10-100 x-rays.
  - Dynamic range (saturation/single photon) is thus only 10:1 to 100:1
- Situation is better for soft x-rays. At 200 eV only 100 electrons per x-ray, dynamic range is 500:1 to 5000:1.
- Radiation damage is a problem for high fluxes of hard x-rays

Deficiencies of CCDs for x-ray imaging: X-ray Coupling

- Polycrystalline phosphor and fiber-optic taper
  - Good x-ray conversion efficiency
  - Good light collection efficiency
  - Poor spatial resolution, >15 microns, determined by phosphor
  - Inflexible, can't change magnification
- Lens coupling with single-crystal phosphor
  - High resolution possible, < 1 micron
  - Trade off of x-ray conversion efficiency for spatial resolution, varies with phosphor thickness.
  - Poor light collection efficiency

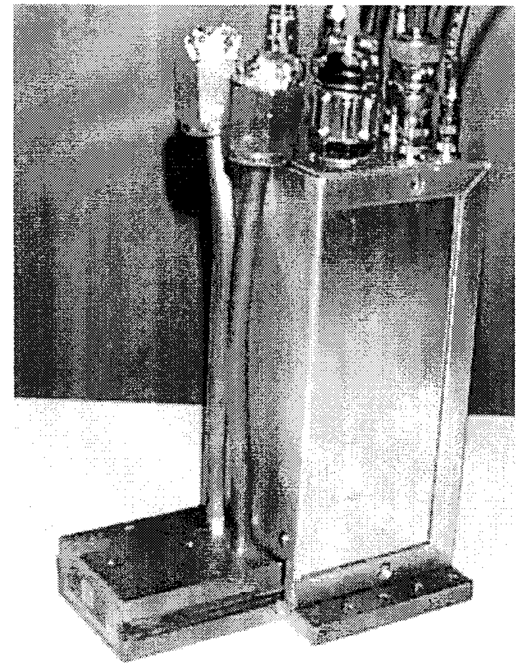
Deficiencies of CCDs for x-ray imaging: Readout Time

- Readout time is 1-5 seconds for high-resolution CCD @16 bits
- This can be 10-1000 times larger than the exposure time for synchrotron experiments
- Multiple-readout ports are available on some chips, rarely implemented on commercial CCD detector systems

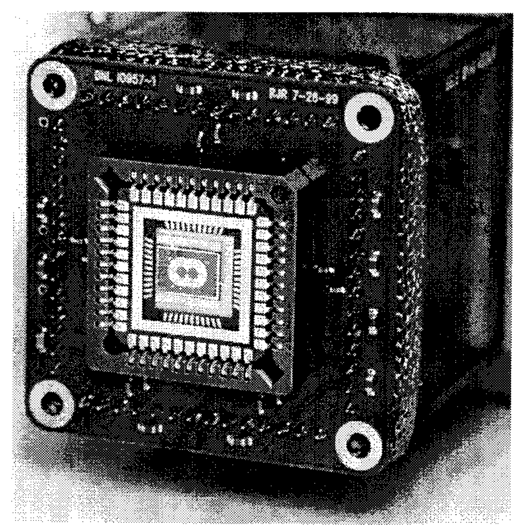
Detector Development for Imaging

- Better CCDs unlikely to be developed by DOE labs
- Better coupling of x-rays to CCDs – structured phosphors, etc. could well be done
- Smaller detectors are being done well now
  - Fast gas proportional counters for scanning microscopy at BNL
  - Few-element Si detector for microscopy at BNL

Detector Development at BNL & Stony Brook



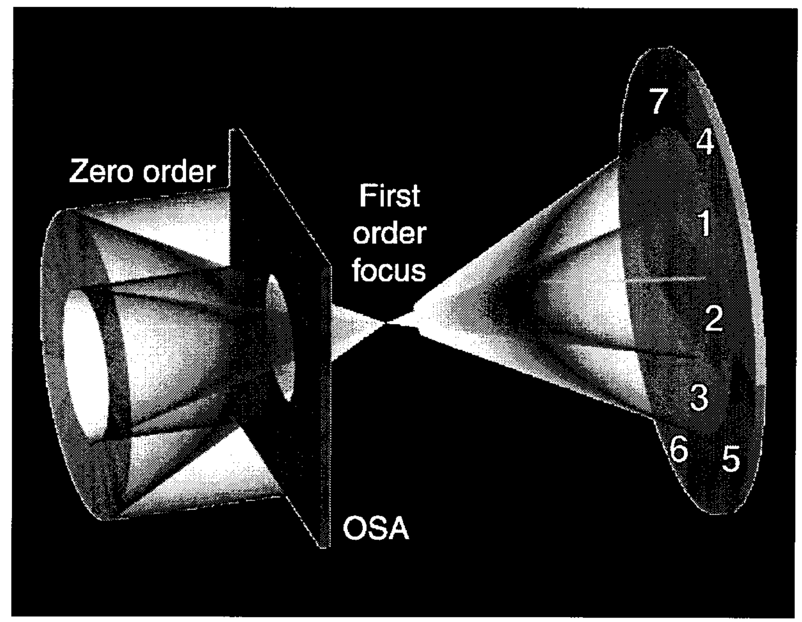
- Continuous flow gas proportional counter
  (Collaboration G. Smith and B. Yu, BNL)
  - Single photon detection
  - No dark count noise
  - Count rate limited to 2 MHz
  - No spatial resolution
  - High maintenance
  - 50 % quantum efficiency (entrance window)



- Integrating silicon detector with segmentation
  (Collaboration P. Rehak and G. DeGeronimo, BNL)
  - No flux limit
  - Flexible imaging modes
  - Analog device – EM pickup
  - Noise is ~ 20 kHz photon count rate equivalent
  - High quantum efficiency (except entrance window)

Soft (250-800 eV) X-ray Scanning Microscopy: Detector Requirements

- Robust and reliable: ~15 different user groups / yr
- Linearity to high count rates: 2 MHz at NSLS; 15 MHz at ALS
- High dynamic range: signals as low as 5 kHz (dark field detection scheme)
- Low noise: ideally single photon sensitivity for highest S/N and elimination of pickup
- High quantum efficiency: Specimen are radiation dose sensitive
- Spatial segmentation matched to STXM geometry (critical illumination, Nomarski, differential phase contrast, dark field)
- Visible light sensitivity?



Thoughts on General Role of DOE Laboratories in Detector Development

- Personal perspective based on 17 years at DOE synchrotrons (NSLS and APS).
- There have been notable successes and some not-so-successful ventures.
  - Has there been any attempt to document the record at DOE labs and understand what types succeed and what do not?
- What is the right role of the labs relative to private industry?
- Labs should embark on detector development for:
  - Small-scale production projects when there is a critical need for the DOE synchrotron community and no prospect of commercialization
  - Larger prototypes only when there is an excellent prospect of commercialization. A single detector is rarely worth developing in the synchrotron field unless rapidly leads to commercialization